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L5: Entry 7 of 48 File: USPT Jul 16, 1996

DOCUMENT-IDENTIFIER: US 5536805 A
TITLE: Mixture of isocyanate-terminated polyurethane prepolymers having good adhesion

BSPR:
In copending Patent application Ser. No. 07/515,113 filed Apr. 27, 1990, now abandoned, the utility of an adhesive, coating and/or sealant formulation consisting essentially of a first isocyanate-terminated polyurethane based on the reaction product of a polyhexamethylene adipate and a polyisocyanate and a second isocyanate-terminated polyurethane based on the reaction product of poly(tetramethylene ether) glycol and a polyisocyanate is described. This formulation has excellent adhesion to a variety of polymeric substrates which previously have not been amenable to adhesive bonding. Such substrates include polystyrene, polycarbonate, polyvinyl chloride, acrylonitrile/butadiene/styrene terpolymers, polyesters, and polymethylmethacrylate.

DEPR:
Hydroxy-functional materials used to prepare the third prepolymer are preferably essentially amorphous. ("Amorphous" is sometimes referred to herein as "glassy".) By "amorphous" or "glassy" it is meant that the hydroxy-functional material exhibits a Tg but lacks a Tm. Preferably the Tg is between about 0.degree. C. and 50.degree. C. and more preferably between about 0.degree. C and 40.degree. C. If the hydroxy-functional material of the third prepolymer is provided in the form of a polyester polyol it may comprise the reaction product of a polyol, for example a diol, and a polyacid, for example, a dicarboxylic acid. The glassy polyester polyol should have a Mn of at least about 1000, preferably between about 1200 and about 7500, and most preferably between about 1500 and about 6000. If the Mn is below 1000, the resultant prepolymer may lack sufficient cohesive strength. When the Mn is above about 7500, the resultant prepolymer is highly viscous and may be difficult to blend and apply at working temperatures.

DEPR:
Other ingredients or adjuvants may be employed with the blends of the invention to impart to or modify particular characteristics of the composition. These ingredients are included in the overall blends or mixtures of the invention rather than being incorporated into the constituent components thereof. The adjuvants should be added only at a level that does not materially adversely interfere with the adhesion of the composition. The adjuvants may comprise up to 50 weight percent of the composition either individually or in combination. For example, chain-extension agents (e.g., short chain polyols such as ethylene glycol or butanediol); fillers (e.g., carbon black; glass, ceramic, metal or plastic bubbles; metal oxides such as zinc oxide; and minerals such as talc, clays, silica, silicates, and the like), thermoplastic resins; plasticizers; antioxidants; pigments; U.V. absorbers; and adhesion promoters such as silanes, and the like may be included to modify set time, open time, green strength build-up, tack, flexibility, adhesion etc.

DEPR:
Table 1 shows that a blend of isocyanate-terminated polyurethane prepolymers based on 1,6-polyhexamethylene adipate, poly(tetramethylene ether) glycol, and an essentially amorphous polyester polyol (examples 6-8) has excellent adhesion to cold rolled steel and abraded aluminum relative to compositions comprising only two of the three prepolymers (examples 2-5). Table 1 further shows that the three component blends (examples 6-8) have adhesion to polystyrene and polymethylmethacrylate comparable to examples 2-3, which examples correspond to copending Patent application Ser. No. 07/515,113 now abandoned. However, examples

6-8 show a significant and surprising increase in plastic adhesion relative to examples 4-5, which examples include a blend of semicrystalline and amorphous prepolymers.

DETL:
TABLE 1 ______________________________________ Example No. 1 2 3 4 5 6 7 8 ______________________________________ 1st Prepolymer.sup.1 100 60 45 60 60 60 60 45 2nd Prepolymer.sup.2 0 40 55 0 0 20 20 20 3rd Prepolymer (a).sup.3 0 0 0 40 0 20 0 0 (b).sup.4 0 0 0 0 40 0 20 35 Peel Adhesion (piw) Abraded 3 20 28 35 32 56 NT 52 aluminum Cold rolled 7 24 15 44 17 54 50 46 steel Polystyrene <1 45 37 28 3 55 41 49 Polymethyl- 36 40 56 6 4 59 45 60 methacrylate ______________________________________ NT = Not tested .sup.1 Prepared from 1,6polyhexamethylene adipate, an essentially semicrystalline polyester polyol (LEXOREZ 113030P from Inolex Chemical Co., hydroxyl number of 30, Mn of 3740). .sup.2 Prepared from a poly(tetramethylene ether) glycol (POLYMEG 2000 from QO Chemical, Inc., hydroxyl number of 56, Mn of 2004). .sup.3 Prepared from an essentially amorphous polyester polyol (DYNACOLL 7110 from Huls America, hydroxyl number of 55, acid number of 10, Mn of 1726). .sup.4 Prepared from an essentially amorphous polyester polyol (DYNACOLL 7111 from Huls America, hydroxyl number of 32, Mn of 3507).

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L5: Entry 3 of 48 File: USPT Jun 8, 1999

DOCUMENT-IDENTIFIER: US 5910540 A
TITLE: Thermoplastic elastomer composition and composite molded product

DEPR:
As a polyester type elastomer, those having a bending modulus according to JIS K-7203 of not more than 10,000 kg/cm.sup.2, particularly preferably not more than 5,000 kg/cm.sup.2, and/or having a melting point by DSC (peak temperature) of not more than 230.degree. C., particularly preferably not more than 220.degree. C. are preferable. Such elastomers can be obtained by polycondensation of an oligomer obtained by esterification or ester exchange reaction of (1) an aliphatic and/or alicyclic diol having 2-12 carbon atoms, (2) an aromatic dicarboxylic acid or an alkyl ester thereof, and (3) a polyalkylene ether glycol having the weight average molecular weight of 400 to 6,000.

DEPR:
As the polyalkylene ether glycol, those having a weight average molecular weight of 400 to 6,000 are used, but a preferable weight average molecular weight is 500 to 4,000, and particularly preferable weight average molecular weight is 600 to 3,000. When the molecular weight is below 400, the resulting copolymer shows inferior block polymerization, and when the weight average molecular weight exceeds 6,000, the physical properties of the resulting polymer are degraded due to the phase separation in the system. Examples of the polyalkylene ether glycol include polyethylene glycol, poly(1,2 and 1,3 propylene ether) glycol, polytetramethylene ether glycol, polyhexamethylene ether glycol, a block or random copolymer of ethylene oxide and propylene oxide, a block or random copolymer of ethylene oxide and tetrahydrofuran, and the like. A particularly preferable example is polytetramethylene ether glycol.

DEPR:
The polyester type copolymer according to the present invention can be (4) a polyester oligomer in which an aliphatic or alicyclic dicarboxylic acid and an aliphatic diol are condensed or (5) a polyester oligomer synthesized from an aliphatic lactone or an aliphatic monool carboxylic acid instead of (3) a polyalkylene ether glycol having a weight average molecular weight of 400 to 6,000.

DEPR:
The polyurethane type thermoplastic elastomer contains a hard segment comprising a diisocyanate and a short chain glycol (ethylene glycol, propylene glycol, 1,4-butanediol, bisphenol A and the like), and a soft segment comprising a diisocyanate and a long chain polyol. The long-chain polyol includes those of a polyether type such as poly(alkylene oxide)glycol having a molecular weight of 400 to 6,000 (for example, polyethylene glycol, poly(1,2 and 1,3 propylene oxide) glycol, poly (tetramethylene oxide) glycol, poly(hexamethylene oxide)glycol and the like), or those of polyester type such as polyalkylene adipate, polycaprolactone, and polycarbonate. Such polyurethane type thermoplastic elastomer is a compound having the structure represented by the following general formula (II). ##STR3## (wherein A represents a hard segment comprising a diisocyanate and a short-chain glycol, B represents a soft segment comprising a diisocyanate and a long-chain polyol, Y represents the residual group of the diisocyanate compound of the urethane bond linking the A segment and B segment.)

DEPR:
It is also possible to blend an additional compounding material including other thermoplastic resins except for the above-mentioned components (A) and (B), or various elastomers, various plasticizers, and various fillers and the like in a

range that does not notably mar the effect of the present invention.

DEPR:
Examples of the plasticizer used as an additional component according to the present invention include a softening agent for a hydrocarbon type rubber having a weight average molecular weight of 300 to 3,000 and polybutene.

DEPR:
These resins can be compounded with various other additives including fillers such as rubber components, talc, calcium carbonate, mica and glass fiber, plasticizers such as paraffin oil, antioxidants, heat stabilizers, light stabilizers, UV absorbing agents, neutralizing agents, slip additives, compatibilizers, lubricants, anti-fogging agents, anti-blocking agents, slipping agents, dispersing agents, coloring agents, antibacterial agents, and fluorescent whiteners.

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| USPT | tetramethylene same adipate | 460 | L1 |

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L8: Entry 2 of 12 File: USPT Apr 14, 1998

DOCUMENT-IDENTIFIER: US 5739325 A
TITLE: Hydrogenolysis of
2,4,6,8,10,12-Hexabenzyl-2,4,6,8,10,12-Hexaazatetracyclo[5.5.0.0.sup.5,9
.0.sup.3,11 ]dodecane

CCXR:
149/92

ORPL:
Chemical Abstracts, 120: 32623e, Propellants and Explosives, "Thermal stability of hexanitrohexaazaisowurtzitane in an Estane formulation", vol. 120, (1994).

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L8: Entry 5 of 12 File: USPT Aug 20, 1996

DOCUMENT-IDENTIFIER: US 5547526 A
TITLE: Pressable explosive granular product and pressed explosive charge

BSPR:
The commercially available explosive/plastic binder granular products for producing pressed charges contain especially polyurethanes as well as fluoropolymers as the plastic binder. Thus, a granular product containing a hexafluoropropylene-vinylidene fluoride polymer known under the trademark "VITON A" and another granular product with a thermoplastic polyurethane binder under the trademark "ESTANE" as the plastic binder are commercially available.

DEPR:
To check the performance capacity of the charge according to the present invention, explosions were carried out with standard hollow charges with a caliber of 96 mm, which were produced from the Octogen/plastic binder granular product according to Example 1. The depth of penetration of the hollow charge spike of this hollow charge into a steel block was evaluated as the performance criterion. At a distance of 768 mm between the hollow charge and the steel block, depths of penetration of between 900 and 1000 mm into the steel block were measured. This corresponds to the results obtained with pressed hollow charges which were produced from the commercially available Octogen/plastic binder granular products PBX N5 (with "Viton A" as the plastic binder) and LX 14 (with "Estane" as the plastic binder), or with a cast hollow charge made from Octol (Octogen/TNT 85/15). Consequently, despite its low sensitivity, the explosive charge according to the present invention has a performance level comparable to that of the prior-art high-performance explosive charges.

DETL:
TABLE IV

Candidate Shaped-Charge Explosives Weight Weight Formulation Binder Percent Explosive Percent Developer

LX-14(N) Estane 4.5 HMX 95.5 LLNL.sup.1 PAX-2 NP/CAB.sup.x 12/8 HMX 80 ARDEC.sup.2 PAX-2A NP/CAB.sup.x 9/6 HMX 85 ARDEC PBXW-9 TYPE II(Q) DOA/Hycar 6/2 HMX 92 NSWCDD PBXW-11 Hycar 3/1 HMX 96 NSWCDD PBXP-31 Silicone 4 HMX 96 MBB.sup.3 ERDCO-301 TNT/Urthn. 15/3 HMX 82 ERDCO.sup.4 OCTOL 85/15 TNT 15 HMX 85 MBB PBXC-129(Q) LM 11 HMX 89 NAWCWD.sup.5 PBXN-110 HTPB 12 HMX 88 NSWCDD

.sup.1 Lawrence Livermore National Laboratory .sup.2 Army Research and Development Engineering Command .sup.3 Messerschmitt Boelkow Blohm .sup.4 Explosive Research and Development Company .sup.5 Naval Air Warfare Center Weapons Division, China Lake, California .sup.x CAB = Cellulose Acetate Butyrate

CCOR:
149/19.2

CCXR:
149/19.91

CCXR:
149/19.92

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L8: Entry 9 of 12 File: USPT Nov 26, 1985

DOCUMENT-IDENTIFIER: US 4555277 A
TITLE: Extrusion cast explosive

BSPR:
U.S. Pat. No. 3,778,319 to Benzinger et al., describes a high energy plastic-bonded explosive of 95% HMX, and 2.5% DNPA/F which is a eutectic mixture of the formal and acetal of 2,2-dinitropropanol as the plasticizer. The plasticizer is a thermoplastic, polyurethane elastomer, manufactured by B. F. Goodrich Company and sold under the trade name of Estane, which is prepared from long chain diols and toluene diisocyanate.

CCOR:
149/19.4

CCXR:
149/88

CCXR:
149/92

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L3: Entry 1 of 2 File: USPT Dec 11, 1990

DOCUMENT-IDENTIFIER: US 4976794 A
TITLE: Thermoplastic elastomer-based low vulnerability ammunition gun propellants

BSPR:
The plasticizer, if used, may be non-energetic, e.g., dioctyl phthalate (DOP), dioctyl adipate (DOA), Santicizer 8 polyester by Monsanto, butanetriol trinitrate (BTTN), trimethylolethane trinitrate (TMETN), polyglycidal nitrate, or nitroglycerine (NG). Generally, if an energetic plasticizer is used, it is used at a low level in order to maintain the low vulnerability properties of the propellant. Other suitable plasticizers include, but are not limited to dibutoxyethyl phthalate (DBEP), dibutoxyethyl adipate (DBEA), chlorinated paraffin, methyl abietate, methyl dihydro-abietate, n-ethyl-o and p-toluene sulfonamide, polypropylene glycol sebacate, dipropylene glycol dibenzoate, di(2-ethyl-hexyl) phthalate, 2-ethyl-hexyl-diphenyl phosphate, tri(2-ethyl-hexyl) phosphate, di(2-ethyl-hexyl)sebacate, Santicizer 409 polyester by Monsanto, tetra-ethylene glycol-di(2-ethyl hexoate), dibutoxyethoxyethyl adipate (DBEEA), N,N,dimethyl oleamide, dibutoxyethyl azelate (DBEZ), dioctyl azelate (DOZ), dibutoxyethoxyethyl glutarate (DBEEG), dibutoxyethyl glutarate (DBEG), polyethylene glycol 400 dilaurate, polyethylene glycol 400 dioleate, dibutoxyethoxyethyl sebacate, dibutoxyethyl sebacate, and trioctyl trimellitate (TOTM).

DETL:
Soft Blocks poly ethylene glycol (PEG) polycaprolactone (PCP) polytetrahydrofuran (PolyTHF) polypropylene glycol (PPG) amorphous polyoxetanes poly(ethylene oxide-tetrahydrofuran) poly(diethylene glycol adipate) polyglycidzyl nitrate polyglycidyl azide (GAP) Hard Blocks polyallyl acrylate polyisobutyl acrylate poly 1,4-cyclohexylenedimethylene formal, trans poly 1,2-cyclopropanedimethylene isophthalate poly decamethylene adipate poly decamethylene azelaate poly decamethylene oxalate poly decamethylene sebacate polyethylene sebacate polyethylene succinate poly hexamethylene sebacate poly 10-hydroxydecanoic acid poly tert-butyl-isotactic poly nonamethylene terephthalate poly octadecamethylene terephthalate poly 3,3-bisethoxymethyl (BEMO) poly pentamethylene terephthalate poly B-propiolactone poly tetramethylene p-phenylenediacetate poly trimethylene oxalate polyethyl vinyl ether polypropyl vinyether poly -p-xylylene adipate poly -p-xylylene sebacate.

CLPR:
5. A propellant composition according to claim 4 wherein said plasticizer is selected from the group consisting of butanetriol trinitrate, trimethylolethane trinitrate and nitroglycerine.

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L5: Entry 16 of 48 File: USPT Dec 11, 1990

DOCUMENT-IDENTIFIER: US 4976794 A
TITLE: Thermoplastic elastomer-based low vulnerability ammunition gun propellants

BSPR:
LOVA gun propellants comprise between about 60 and about 85 wt. percent of a high-energy oxidizer particulates and between about 15 and about 40 wt. percent of an elastomeric, thermoplastic binder system. The thermoplastic elastomer of the binder system has at least one block which is amorphous at room temperature, e.g., in the range of about 20.degree. C. to about 25.degree. C. and at least one block which is crystalline at room temperature. It is generally necessary that in the block copolymer molecule, there be at least a pair of crystalline blocks flanking an amorphous block, whereby a thermoplastic network may be formed. The crystalline hard blocks preferably melt in a temperature range of between about 70.degree. C. and about 105.degree. C. This temperature range allows processing at temperatures which do not decompose the nitramine fillers. At the same time, in this temperature range, the binder retains good mechanical properties at about 63.degree. C., considered to be the upper use temperature of LOVA gun propellants. The binder system may contain up to about 80 wt. percent of an energetic or non-energetic plasticizer, the plasticizer comprising up to about 35 wt. percent of the LOVA gun propellant composition as a whole.

BSPR:
The plasticizer, if used, may be non-energetic, e.g., dioctyl phthalate (DOP), dioctyl adipate (DOA), Santicizer 8 polyester by Monsanto, butanetriol trinitrate (BTTN), trimethylolethane trinitrate (TMETN), polyglycidal nitrate, or nitroglycerine (NG). Generally, if an energetic plasticizer is used, it is used at a low level in order to maintain the low vulnerability properties of the propellant. Other suitable plasticizers include, but are not limited to dibutoxyethyl phthalate (DBEP), dibutoxyethyl adipate (DBEA), chlorinated paraffin, methyl abietate, methyl dihydro-abietate, n-ethyl-o and p-toluene sulfonamide, polypropylene glycol sebacate, dipropylene glycol dibenzoate, di(2-ethyl-hexyl) phthalate, 2-ethyl-hexyl-diphenyl phosphate, tri(2-ethyl-hexyl) phosphate, di(2-ethyl-hexyl)sebacate, Santicizer 409 polyester by Monsanto, tetra-ethylene glycol-di(2-ethyl hexoate), dibutoxyethoxyethyl adipate (DBEEA), N,N,dimethyl oleamide, dibutoxyethyl azelate (DBEZ), dioctyl azelate (DOZ), dibutoxyethoxyethyl glutarate (DBEEG), dibutoxyethyl glutarate (DBEG), polyethylene glycol 400 dilaurate, polyethylene glycol 400 dioleate, dibutoxyethoxyethyl sebacate, dibutoxyethyl sebacate, and trioctyl trimellitate (TOTM).

DEPR:
Table 1 below summarizes various properties of LOVA gun propellants prepared using different thermoplastic elastomeric binder systems, including mixing conditions, extrusion conditions, mechanical and physical properties and burn rates. In each case, the composition is 78% RDX, 22% binder system. The third composition from the left has a binder system which includes 20% by weight of a non-energetic plasticizer, dioctyl phthalate (DOP). The fourth polymer is of the type reported in above-identified U.S. patent application Ser. No. 06,925,660 as being an ABA block polymer wherein poly(3,3-bix(azidomethyl)oxetane) (BAMO) forms the crystalline A blocks and wherein the B block is a copolymer of poly(3,3-bis(azidomethyloxetane/3-azidomethyl-3-methyloxetane) (BAMO/AMMO).

DETL:
TABLE I

PES
LRG269 LRG269B Polymer PDEGA Santicizer 8 DOP (4:1) B-B/A-B

Rheocord 40 Test (78% RDX) LT035 LT033 LT051 LT049 Peak Torque, m-g 590 416 1255 971 Peak Temperature, .degree.C. 116.degree. 114.degree. 128.degree. 119.degree. Extrusion (EX87) 0707-2 0629 0930-2 0921-2 600 psi Barrel T, .degree.C. 89.degree. (750 psi) 95.degree. 112.degree. 85.degree. Die T, .degree.C. 80.degree. 85.degree. 99.degree. 78.degree. DSC (10.degree. C./min, N.sub.2) Tg, .degree.C. -44.degree. -54.degree. -35.degree. -41.degree. Tm, .degree.C. +79.degree. +93.degree. +120.degree. +93.degree. 63.degree. C. Slump. Compressibility, % 2.2 19 1.9 2.2 60 Min Creep, % 1.6 17 0.3 1.2 DMA (5.degree. C./Min) Tg, .degree.C. -33.degree. -39.degree. -64.degree. -24.degree. E' @ -40.degree. C., MPa 568 508 343 763 0.degree. 224 89 201 315 +20.degree. 151 55 162 195 +40.degree. 55 9 99 118 Tensiles @ 25.degree. C. (0.1 in/min) Modulus, psi 14,000 6000 25,300 21,000 Stress, psi 234 59 460 235 Strain, % 2.2 1.1 2.0 1.3 Burn Rate @ 11,000 psi, in/sec 0.85 1.10 0.76 1.88 26,000 psi, in/sec 289 4.09 2.09 4.82

DETL:

Soft Blocks poly ethylene glycol (PEG) polycaprolactone (PCP) polytetrahydrofuran (PolyTHF) polypropylene glycol (PPG) amorphous polyoxetanes poly(ethylene oxide-tetrahydrofuran) poly(diethylene glycol adipate) polyglycidzyl nitrate polyglycidyl azide (GAP) Hard Blocks polyallyl acrylate polyisobutyl acrylate poly 1,4-cyclohexylenedimethylene formal, trans poly 1,2-cyclopropanedimethylene isophthalate poly decamethylene adipate poly decamethylene azelaate poly decamethylene oxalate poly decamethylene sebacate polyethylene sebacate polyethylene succinate poly hexamethylene sebacate poly 10-hydroxydecanoic acid poly tert-butyl-isotactic poly nonamethylene terephthalate poly octadecamethylene terephthalate poly 3,3-bisethoxymethyl (BEMO) poly pentamethylene terephthalate poly B-propiolactone poly tetramethylene p-phenylenediacetate poly trimethylene oxalate polyethyl vinyl ether polypropyl vinyether poly -p-xylylene adipate poly -p-xylylene sebacate.

CLPR:
1. A low vulnerability ammunition gun propellant composition comprising from about 60 to 85 wt. percent of particulates of a high-energy oxidizer and between about 15 wt. percent and about 40 wt. percent of a thermoplastic, elastomeric binder system, said binder system being substantially free of metallic particulates and materials which leave a solid residue, said binder system comprising a non-cross-linked, thermoplastic, elastomeric polymer in which at least one pair of crystalline A blocks flanks at least one amorphous B block and from 0 to about 80 wt. percent of a plasticizer, wherein said non-cross-linked, elastomeric polymer comprises crystalline polyester A blocks and an amorphous polyester B block.

CLPR:
2. A propellant composition according to claim 1 which includes a plasticizer which is non-energetic.

CLPR:
3. A propellant composition according to claim 2 wherein said non-energetic plasticizer is dioctyl phthalate.

CLPR:
4. A propellant composition according to claim 1 which includes a plasticizer which is energetic.

CLPR:
5. A propellant composition according to claim 4 wherein said plasticizer is selected from the group consisting of butanetriol trinitrate, trimethylolethane trinitrate and nitroglycerine.

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L8: Entry 11 of 12 File: USPT Aug 18, 1981

DOCUMENT-IDENTIFIER: US 4284442 A
TITLE: Castable TNT compositions containing a broad spectrum preformed thermoplastic polyurethane elastomer additive

DEPR:
A lacquer of Estane was prepared by dissolving 3 parts of Estane 5702 in 40 parts of methyl ethyl ketone. Estane 5702 is believed to be a thermoplastic, essentially fully reacted polyurethane elastomer containing essentially no free isocyanate groups, prepared from 4,4'-diphenylmethane diisocyanate, poly (tetramethyleneadipate) glycol and 1,4-butanediol, manufactured by the B. F. Goodrich Co. Estane 5702 has the following physical properties:

DEPR:
The Estane lacquer was stirred into a slurry of 97 parts of RDX, Class A, average particle size about 175 microns, in 100 parts of distilled water containing 0.1 part of polyvinylpyrrolidone of m.w. 90,000. 300 Parts of cold water (8.degree. C.) were added with agitation to the mixture thus obtained, thereby cooling the mixture to 14.degree. C. and precipitating the Estane as a coating on the finely divided RDX. The precipitate was separated by filtration and the filter cake was washed with two portions each of 100 parts of cold water (8.degree. C.) and dried on a tray at 100.degree. C. The dry, coated RDX was obtained as free-flowing granules of bulk density 0.62 gr/cc.

DEPR:
40 parts of technical grade TNT (solidification point 80.2.degree. C.) were heated with agitation to 85.degree.-90.degree. C. 60 parts of the product obtained in A, comprising 58.2 parts of RDX coated with 1.8 parts of Estane 5702, were preheated to 90.degree. C. and stirred into the molten TNT. The resulting mixture was agitated at 85.degree.-90.degree. C. for several minutes until the RDX was uniformly dispersed, and then poured into cylindrical containers and allowed to solidify.

DEPR:
The solidified cast product thus obtained possessed excellent homogeneity, heat stability and freedom from exudation, viz. 0.05% exudation after 5 days at 70.degree. C. versus 0.18% exudation under similar condition when the Estane was omitted. Other advantageous properties imparted to the cast explosive by the Estane includes the following:

DEPR:
Similar results were obtained by replacing Estane 5702 with Plastothane.TM. 430, which is said to be a fully reacted polyurethane elastomer containing no free isocyanate groups having the following physical properties:

DEPR:
Composition B, a mixture of 60 parts RDX, 40 parts TNT and 1 part wax, was heated to 85.degree.-90.degree. C. until the TNT was completely melted. The wax separated as a supernatant layer on the melt and side of the container. 0.12 Part of Estane 5702 was added to the melt with agitation and the resulting mixture was agitated at 85.degree.-90.degree. C. for about 30 minutes. The incorporation of the Estane unexpectedly eliminated the wax segregation from the melt. The melt was poured into molds and allowed to cool and solidify. The solid cast explosive composition thus obtained possessed similar properties to those of the cast product of example 1.

DEPR:

The control cast Composition B (containing no Estane additive) contained an oriented structure of large TNT crystals.

DEPR:
Cast explosive charges were prepared by incorporating Estane 5702, alone or together with alpha cellulose powder, into a mixture of 60 parts RDX and 40 parts TNT at 85.degree.-90.degree. C., agitating the resulting composition at 85.degree.-90.degree. C. until homogeneous and pouring the molten composition into molds wherein it was allowed to cool and solidify.

DEPR:
Control compositions were similarly prepared by employing standard wax in place of Estane 5702.

DEPR:
To achieve a solid cast TNT explosive composition possessing a fine random oriented TNT crystal structure and other advantageous properties noted above, it is preferred to incorporate about from 0.05% to 10% and especially from 0.1% to 5% by weight of the preformed, fully reacted thermoplastic polyurethane additive, such as Estane, based on the TNT content of the explosive composition. The incorporation of substantially more than 10% of the polyurethane additive by weight of the TNT is less preferred, since it dilutes the explosive power, increases the melt viscosity excessively and produces a cast product, which although characterized by the fine random TNT crystal structure, tends to break along massive cleavage planes when subjected to breaking.

DEPV:
(1) The Estane modified solid TNT matrix was characterized by highly desirable fine, random oriented TNT crystals similar to that obtained when hexanitrostilbene additive is employed.

DEPV:
(5) The cast possessed substantially better impact resistance. The Estane eliminated the need for the wax desensitizer conventionally employed.

DETL:

Minimum Velocity Maximum Velocity to cause explosion for no explosion ft/sec ft/sec
Composition B (60/40/1 RDX/TNT/wax) 2972 2856 pressed billet Composition B cast billet typical range of 2700-3300* results* Composition B + 0.12% Estane cast billet 3080 3037
*The wax employed as a desensitizer, unlike Estane 5702, is insoluble in the TNT and hence products cast product of varying uniformity and explosive properties.

DETL:
Compression Strength at 70.degree. F.
Additive (1/2 in. .times. 1/2 in. to 60/40 RDX/TNT cylinders) psi
1% Wax 436 1% Wax + 0.24% .alpha.-cellulose.sup.(1) 911 1% Estane 5702 977 1% Estane 5702 + 0.24% .alpha.-cellulose.sup.(1) 1117 1.8% Estane 5702 1049 1.8% Estane 5702 + 0.24% .alpha.-cellulose.sup.(1) 1318 .sup.(1)
The .alpha.cellulose used was finely dividedcellulose made from wood pulp and marketed by the Brown Co. under the trade name SolkaFloc, grade SW40 having the following properties: average fiber length, 100-140 microns; apparent density range, 6-12 lbs. per cu. ft.; pH, 10% aqueous suspension, 5.5-6.5

CCOR:
149/19.4

CCXR:
149/105

CCXR:
149/18

CCXR:
149/92

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L8: Entry 10 of 12 File: USPT Apr 20, 1982

DOCUMENT-IDENTIFIER: US 4325759 A
TITLE: Preparation of TNT-thermoplastic polymer granules readily soluble in a TNT melt

BSPR:
The aforementioned application discloses the incorporation of a preferred thermoplastic polymethane elastomer, Estane 5702 (manufactured by the B.F. Goodrich Co. and believed to be the reaction product of 4,4'-diphenylmethane diisocyanate, poly (tetramethyleneadipate) glycol and 1,4-butanediol) in a TNT-based castable explosive composition to produce a modified Composition B explosive consisting of 59.5 parts RDX, 0.5 part Estane 5702 and 40 parts TNT. Estane 5702 and other thermoplastic polyurethane elastomer additives generally dissolve slowly with difficulty in molten TNT in which they have limited solubility. The present method for dissolving the elastomer additive in a TNT melt comprises precoating the elastomer on the crystalline RDX particles by precipitating the elastomer from a solution thereof in an organic solvent, which spreads the elastomer over a large surface and thereby increases the rate of solution of the elastomer in the molten TNT. However, even when thus coated or mixed with the RDX particles, the elastomer dissolves rather slowly in molten TNT, requiring several hours to effect complete solution. Also the method of coating the elastomer on the RDX particles is not entirely satisfactory, since it entails a number of operations, which increase the cost of manufacture and limit the capacity for producing the cast shell filler, which is particularly serious in times of emergency.

BSPR:
To alleviate this problem, an attempt has been made to prepare a concentrate consisting of TNT and the elastomer, e.g. about 2 parts TNT and 1 part Estane 5702, which could be incorporated directly in molten TNT and perhaps dissolve more rapidly therein due to the substitution of soluble TNT in place of RDX, which is essentially insoluble in molten TNT. The method involved dissolving the mixture of TNT and Estane in acetone and evaporating the solvent solution to dryness. Such attempts were unsuccessful, since the product was obtained in the form of a hard, gritty product which dissolved slowly in molten TNT.

BSPR:
In accordance with the present invention there is provided a simple and efficient process for producing a concentrate consisting essentially of TNT and a thermoplastic organic polymer, such as Estane, in the form of granules, which dissolve rapidly in molten TNT.

BSPR:
The process of the present invention comprises dissolving a mixture of TNT and the thermoplastic organic polymer, such as Estane 5702, in a water-soluble organic solvent in which the TNT and the polymer are soluble, and precipitating the mixture from the solution by dilution with water. In a preferred embodiment of the present process, the solution is diluted with a relatively large amount of water, preferably at least about five parts by volume of water per part of the organic solvent, with vigorous agitation, whereby the TNT-polymer concentrate is precipitated in the form of granules, which can be readily separated from the liquor by filtration and dissolve quickly in molten TNT. For optimum results the dilution water is employed in an amount which is sufficient to dissolve the solvent substantially completely. Advantageously, a small amount, e.g. 0.01% to about 1%, of a dispersing agent or protective colloid, such as gum arabic, arabinogalactan, and sodium dodecylbenzensulfonate is added to the dilution water, since it has a beneficial effect on the uniformity and solubility rate in

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DOCUMENT-IDENTIFIER: US 4102868 A
TITLE: Composition and process for the preparation of segmented polyester-urethane polymers

BSPR:
One embodiment of the invention comprises segmented elastoplastic urethane polymer prepared by heating at 225.degree. C. or higher a slurry consisting essentially of (a) about 40 to about 80 weight percent of at least one liquid dihydroxy polyester of the formula ##STR1## or liquid polyether of the formula H(OR.sub.2).sub.n OH wherein R is alkylene of 2 to 10 carbon atoms or said alkylene interrupted by oxygen, R.sub.1 is alkylene of 2 to 8 carbon atoms, R.sub.2 is alkylene of 2-4 carbon atoms and n is 2 to about 50, (b) about 60 to about 10 weight percent hydroxy-terminated poly(tetramethyleneterephthalate) or hydroxy-terminated poly(ethyleneterephthalate) having a molecular weight from about 1000 to about 12,000, and (c) about 2 to about 15 weight percent organic diisocyanate, wherein the NCO/OH ratio is one or more, for a time sufficient to form essentially linear segmented urethane polymer. Said segmented elastoplastic urethane polymers exhibit lower hysteresis than polymers of similar composition prepared in an inert solvent. The distribution of polymer segments of polymers prepared by the above-described slurry process is believed to be different from the distribution of polymer segments of polymers prepared by a solvent process which difference in distribution probably accounts for the improved properties of the polymers. Polymers in which (a) is a polyester are preferred. More preferred polymers exhibiting lower hysteresis consist essentially of about 50 to about 75 weight percent segments derived from at least one dihydroxy-terminated polyester having a melting point of 50.degree. C. or less, and a molecular weight of from about 1000 to about 12,000, preferably about 1800 to about 6000, and about 50 to about 25 weight percent segments derived from poly(tetramethyleneterephthalate) having a molecular weight of about 1800 to about 6000. The extreme of the ranges cannot occur simultaneously. For example, in a polymer containing 60 weight percent of high melting-polyester, the amount of low-melting polydiol must be less than 40 weight percent so that enough organic diisocyanate is present to form the segmented polymer, in which case the combined weights of (a) and (c) totals 40 weight percent.

BSPR:
Any dihydroxy polyether or dihydroxy polyester having a molecular weight of from about 500 to about 12,000 which is a liquid at a temperature of 100.degree. C. or below, preferably, a liquid at room temperature, is satisfactory for carrying out the process of the invention. Preferred polyesters or polyethers have a glass transition temperature (Tg) below room temperature. The liquid polyesters are prepared by conventional techniques by esterfication of one or more dicarboxylic acid with one or more dihydric alcohol (MW 300 or less) or by ester interchange of one or more dicarboxylic acid alkyl ester with one or more dihydric alcohol. Liquid polyethers and polyesters having a molecular weight between about 1800 and about 5500 are preferred. Examples of suitable dihydroxytermined liquid polyesters are poly-2,2-dimethylpropylene adipate (PDMPA), polytetramethylene adipate (PTMA), poly-1,3-butylene adipate (P-1,3-BA), polyoxydiethylene adipate (PODEA), poly-1,2-propylene adipate (PPA), polyoxydi-1,2-propylene adipate (POPA), polytriethylene glycol adipate (PTEGA), polytetraethylene glycol adipate (PTTGA), polyethylene succinate (PES), polytetramethylene adipate-co-succinate (PTMAS), polyethylene-co-propylene adipate (PEPA), polyethylene-co-butylene adipate (PEBA), polypropylene-co-tetramethylene adipate (PPMA), poly-tetramethylene-2-ethyl-3-n-propyl trimethylene adipate-co-glutarate (PBGA), polyethylene adipate (PEA), poly-(2-ethyl-3-n-propyl)propylene glutarate (PEPG), polyoxytriethylene succinate (POES), polytetramethylene succinate (PTMS),

polycaprolactone (PCL), polytetramethylene azelate (PTMAZ), polytetramethylene sebacate (PTMSE), poly-1,3-butylene sebacate (P-1,3-BSE), poly-1,3-butylene succinate (P-1,3-BS), polytetramethylene glutarate (PMG), polypentamethylene glutarate (PPMG), poly-2,2-dimethylpropylene glutarate (PDMPG), and poly-1,2-propylene glutarate (PPGL). Additional examples of satisfactory liquid polyesters (which may be selected on the basis of Tg are shown in Table I, pages 31-59, Polyesters, Korshak and Vinogradova, Pergamon Press and Table II, pages 70-72, Encyclopedia of Polymer Science and Technology, Vol. 11, the disclosures of which are herein incorporated by reference. Mixtures of two or more different liquid polyesters may be used with good results.

BSPR:
Any high-melting dihydroxy polyester having a molecular weight of from about 500 to about 12,000 which is a solid at a temperature of 200.degree. C. or above, preferably, a solid at 225.degree. or above, is satisfactory for carrying out the process of the invention. The high-melting polyesters are prepared by the conventional techniques indicated as being suitable for the liquid polyesters. High-melting polyesters having a molecular weight between about 1500 and about 8000 are preferred with molecular weights between about 2000 and about 6000 being more preferred. Examples of suitable dihydroxy-terminated high-melting polyesters are polyethylene terephthalate (PET), polytetramethylene terephthalate (PTMT), polytetramethylene isophthalate (PTMI), poly-(1,4-cyclohexane dimethylene)-co-tetramethylene terephthalate (PCHDMT), polytetramethylene naphthalene-2,6-dicarboxylate (PTMND), polyethylenenaphthalene-2,6-dicarboxylate (PEND), polyethylene diphenylmethane-4,4'-dicarboxylate (PEDPM), polyethylene-1,1'-diphenoxybutane-4,4'-dicarboxylate (PEDPB), polytrimethylene diphenylmethane-4,4'-dicarboxylate (PPMDP), polytetramethylene diphenyl-4,4'-dicarboxylate (PTMDP), polyhexamethylene diphenyl-4,4'-dicarboxylate (PHMDP), poly-1,4-cyclohexane succinate (PCHS), poly-1,4-cyclohexane glutarate (PCHG), and poly-1,4-cyclohexane adipate (PCHA). Additional examples of satisfactory high-melting polyesters (which may be selected on the basis of melting point) are shown in the references cited above in the paragraph on liquid polyesters. Preferred high-melting polyesters develop substantial crystallinity in a reasonable period of time. Polyalkylene terephthalates are preferred. Mixtures of one or more high-melting polyesters may be used, if desired.

BSPR:
The polymers prepared by the process of the invention are useful for making a variety of articles such as tires, hoses, belts, gaskets, moldings and molded parts. Articles may be prepared by any suitable procedure but due to thermoplasticity of the polymers articles are readily prepared by extrusion, injection molding and compression molding techniques. The polymers may be modified by incorporating conventional compounding ingredients therein such as, reinforcing agents, fillers, extenders, pigments, antidegradants, fire retardants, plasticizers, resins or other polymers.

DEPR:
Segmented elastoplastic urethane polymers comprising poly(tetramethylene terephthalate), hexamethylene diisocyanate and low-melting polyesters of poly(1/1 ethylene-co-1,4-butylene adipate), poly(1,2-propylene adipate) and poly(1,3-butylene adipate) are shown in Tables 2, 3 and 4, respectively. The experiments with poly(1/1 ethylene-co-1,4-butylene adipate) of Table 2 are carried out with salt bath temperatures of 278.degree.-298.degree. C. for times of from 4 to 8.5 minutes with the majority of the experiments being carried out at temperatures of 284.degree.-293.degree. C. for times of 5 to 7 minutes. The experiments with poly(1,2-propylene adipate) of Table 3 are carried out with salt bath temperatures of 260.degree.-273.degree. C. for times of about 6-18 minutes with the majority of the experiments being carried out at temperatures of 264.degree.-270.degree. C. for times of about 7 to 12 minutes. The experiments with poly(1,3-butylene adipate) of Table 4 are carried out with salt bath temperatures of 269.degree.-283.degree. C. for times of about 5-15 minutes with the majority of the experiments being carried out at temperatures of 275.degree.-280.degree. C. for times of about 7-12 minutes.

CLPR:
24. The polymer of claim 23 in which the molecular weights of (a) and (b) are between about 1800 to about 6000.